

CERTIFICATE OF MAILING BY FIRST CLASS MAIL (37 CFR 1.8)

Applicant(s): Takamasa HARADA et al.

Docket No.

2002DE301

Serial No.

10/500,194

Filing Date

June 24, 2004

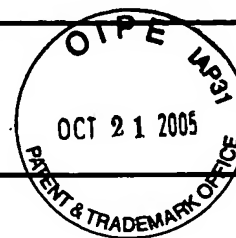
Examiner

To Be Assigned

Group Art Unit

To Be Assigned

Invention: NANOCOMPOSITE MATERIALS FOR THE PRODUCTION OF FILMS
WITH A REFRACTIVE INDEX GRADIENT



I hereby certify that this English Language Translation of JP 09-222504 - 13 Pages
(Identify type of correspondence)

is being deposited with the United States Postal Service as first class mail in an envelope addressed to: The
Commissioner of Patents and Trademarks, Washington, D.C. 20231-0001 on October 19, 2005
(Date)

MARIA T. SANCHEZ

(Typed or Printed Name of Person Mailing Correspondence)


(Signature of Person Mailing Correspondence)

Note: Each paper must have its own certificate of mailing.

PATENT ABSTRACTS OF JAPAN

(11)Publication number : 09-222504

(43)Date of publication of application : 26.08.1997

(51)Int.Cl.

G02B 1/11

B32B 7/02

B32B 9/00

B32B 27/00

B32B 27/16

(21)Application number : 08-052600

(71)Applicant : DAINIPPON PRINTING CO LTD

(22)Date of filing : 16.02.1996

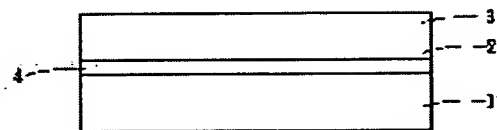
(72)Inventor : OOTA YURIE
NAKAMURA NORINAGA
TSUCHIYA MITSURU

(54) ANTIREFLECTION FILM

(57)Abstract:

PROBLEM TO BE SOLVED: To cost effectively obtain an antireflection film of which the low-refractive index layer has an excellent adhesion property to a high refractive index hard coating layer without using costly and intricate equipment, etc., by forming the high-refractive index hard coating layer of the antireflection film as a specific gel film.

SOLUTION: The high-refractive index layer of the antireflection film constituted by laminating the hard coating layer 2 having a high refractive index and the low-refractive index layer 3 on a transparent base material film 1 is composed of the gel film which is formed from a sol liquid contg. the oxide sol of titanium or tantalum and a reactive org. silicon compd. in a liquid medium and has a refractive index of ≥ 1.65 . Any films are usable as the transparent base material film, insofar as the films have transparency. Uniaxially or biaxially stretched polyester is used adequately in terms of excellent transparency and heat resistance and absence of optical anisotropy. The metal oxide sol of the titanium or tantalum is preferably formed by hydrolyzing the respective metal alkoxides.



LEGAL STATUS

[Date of request for examination]

07.02.2003

[Date of sending the examiner's decision of rejection]

[Kind of final disposal of application other than the
examiner's decision of rejection or application converted
registration]

[Date of final disposal for application]

[Patent number]

[Date of registration]

[Number of appeal against examiner's decision of rejection]

[Date of requesting appeal against examiner's decision of rejection]

[Date of extinction of right]

Copyright (C); 1998,2003 Japan Patent Office

* NOTICES *

JPO and NCIPI are not responsible for any damages caused by the use of this translation.

1. This document has been translated by computer. So the translation may not reflect the original precisely.
2. **** shows the word which can not be translated.
3. In the drawings, any words are not translated.

CLAIMS

[Claim(s)]

[Claim 1] the acid-resisting film which comes to carry out the laminating of the rebound ace court layer and low refractive-index layer which have a high refractive index on a transparence base material film -- setting -- the above-mentioned high refractive-index layer -- a solvent -- the acid-resisting film characterized by consisting of with a refractive indexes of 1.65 or more formed in the inside of the body from the sol liquid containing titanium, or the oxide sol and the reactant organosilicon compound of a tantalum gel film.

[Claim 2] The acid-resisting film according to claim 1 with which sol liquid contains the reactant organosilicon compound 0.1 - 50 weight sections per oxide sol (solid content) 100 weight section of titanium or a tantalum.

[Claim 3] The acid-resisting film according to claim 1 whose reactant organosilicon compound is the organosilicon compound which has two or more functional groups which can be hardened by heat and/or ionizing radiation, or its partial hydrolysate.

[Claim 4] The acid-resisting film according to claim 1 with which functional separation of the rebound ace court layer which has a high refractive index is carried out at the rebound ace court layer and the high refractive-index layer.

[Claim 5] The acid-resisting film according to claim 1 with which a detailed irregularity configuration is formed in a front face, and anti-dazzle property is given.

[Claim 6] The acid-resisting film according to claim 1 which titanium or the oxide sol of a tantalum dissolves in the organic solvent suitable for spreading of a metal alkoxide, adds the water of a constant rate, hydrolyzes, and is prepared.

[Claim 7] The acid-resisting film according to claim 1 whose low refractive-index layer is the thin film of MgF₂ or SiO₂ formed by vacuum evaporatio, sputtering, and the plasma-CVD method.

[Claim 8] The acid-resisting film according to claim 1 whose low refractive-index layer is the thin film of SiO₂ formed from the SiO₂ sol solution.

[Translation done.]

* NOTICES *

JPO and NCIPI are not responsible for any damages caused by the use of this translation.

- 1.This document has been translated by computer. So the translation may not reflect the original precisely.
- 2.**** shows the word which can not be translated.
- 3.In the drawings, any words are not translated.

DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Field of the Invention] This invention relates to the front face of the polarizing plate used for various displays, such as a word processor, a computer, television, and a plasma display panel, and a liquid crystal display, the sunglass lens which consists of transparent plastics, and the acid-resisting film which was [whenever] excellent in acid resisting of front faces, such as windowpanes, such as covering of optical lenses, such as a with glass lens and a finder lens for cameras, and various instruments, an automobile, and an electric car.

[0002]

[Description of the Prior Art] On the display of mirror on a curved road, a reflector glass, goggles, a windowpane, a personal computer, a word processor, a plasma display, etc., and other various commercial displays, conventionally Transparence substrates, such as glass and plastics, are used and it lets these transparence substrates pass. A body and an alphabetic character, When observing vision information, such as a graphic form, or when the image from a reflecting layer was observed through a transparence substrate by the mirror, there was a problem that the front face of these transparence substrates reflected by outdoor daylight, and vision information on internal could not be in sight easily.

[0003] As an approach of preventing an echo of such a transparence substrate, there was the approach of forming the thin film of MgF₂ of about 0.1 micrometers of thickness or SiO₂ grade in the front face of transparence substrates, such as the approach of applying an acid-resisting coating to the front face of glass or plastics and a glass plastics base material, by gaseous-phase methods, such as vacuum evaporatio, and sputtering, a plasma-CVD method, through a rebound ace court layer if needed conventionally.

[0004]

[Problem(s) to be Solved by the Invention] However, carry out coating of the ionizing-radiation hardening mold resin to the front face of plastics, such as a plastic lens, and it considers as a rebound ace court layer. By the approach of forming the thin film of MgF₂ of about 0.1 micrometers of thickness, or SiO₂ grade by vacuum evaporatio, and forming an acid-resisting film on the obtained rebound ace court layer The adhesion of the vacuum evaporatio thin film of MgF₂ to a rebound ace court layer or SiO₂ grade is inadequate, and when an acid-resisting film is repeated and is made crooked, there is a problem of a crack going into these thin films, or a thin film exfoliating.

[0005] as the approach of obtaining the thin film of the quality which was excellent by the applying method in recent years -- inorganic or an organic ultrafine particle -- acidity -- and -- or the dispersion liquid distributed in the alkali water solution are applied on a substrate, and the approach of calcinating is proposed. According to this manufacture approach, in respect of mass production method or facility cost, it is advantageous, but Since the baking process in an elevated temperature is needed in a production process, membranes cannot be formed to a plastics base material, Moreover, by the difference of whenever [contraction-with substrate and spreading film] etc., the homogeneity of a coat is not enough, and in comparison with the thin film obtained by the gaseous-phase method, in the adhesion over a base material etc., the engine performance is still inferior, and heat treatment takes long duration (for example, above for dozens of minutes), and it has the fault referred to as inferior to productivity. Therefore, the object of this invention is offering the acid-resisting film which has the adhesion in which the low refractive-index layer of the maximum surface was excellent to the rebound ace court layer in an acid-resisting film.

[0006]

[Means for Solving the Problem] The above-mentioned object is attained by the following this inventions. namely, the acid-resisting film with which this invention comes to carry out the laminating of the rebound ace court layer and low refractive-index layer which have a high refractive index on a transparence base material film -- setting -- the above-mentioned high refractive-index layer -- a solvent -- it is the acid-resisting film characterized by consisting of with a refractive indexes of 1.65 or more formed in the inside of the body from the sol liquid containing titanium, or the oxide sol and the reactant organosilicon compound of a tantalum gel film. According to this invention, it can provide economically by forming the high refractive-index rebound ace court layer of an acid-resisting film as with a refractive indexes of 1.65 or more gel film into a liquid medium from the sol liquid containing titanium, or the oxide sol and the reactant organosilicon compound of a tantalum, without using the acid-resisting film which has the adhesion in which the low refractive-index layer was excellent to the high refraction rebound ace court layer for an expensive and complicated facility etc.

[0007]

[Embodiment of the Invention] Next, the gestalt of operation is mentioned and this invention is further explained to a detail. Drawing 1 is drawing showing the cross section of one example of the acid-resisting film of this invention in illustration. The acid-resisting film of this example is the example which carried out the laminating of the rebound ace court layer 2 which has a high refractive index on the transparence base material film 1, and the low refractive-index layer 3, and the sign 4 in drawing is the glue line or primer layer by which a laminating is carried out if needed. The example shown in drawing 2 is an example which carried out functional separation of the rebound ace court layer 2 which has a high refractive index in the example shown in above-mentioned drawing 1 at the rebound ace court layer 5 and the high refractive-index layer 6 formed on it. The example shown in drawing 3 is an example which prepared the detailed irregularity configuration in the front face, and gave anti-dazzle property to the acid-resisting film in the example shown in above-mentioned drawing 1.

[0008] In this invention as the above-mentioned transparence base material film Which film is sufficient as long as it is a transparent film. For example, a triacetyl cellulose film, a diacetyl cellulose film, An acetate-butylate cellulose film, a polyether sulphone film, The Pori acrylic resin film, a polyurethane system resin film, polyester film, Although a polycarbonate film, a polysulfone film, a polyether film, a trimethyl pentene film, a polyether ketone film, an acrylonitrile (meta) film, etc. can be used One shaft or biaxial-stretching polyester is excellent in transparency and thermal resistance, and is suitably used at the point which does not have an anisotropy optically. As for the thickness, a 8 micrometers - about 1000 micrometers thing is usually used suitably.

[0009] In the example of drawing 1, the rebound ace court layer which has the high refractive index formed in the field of the above-mentioned transparence base material film is the with a refractive indexes of 1.65 or more formed into the liquid medium from the mixture of the sol liquid containing titanium, or the oxide sol and the reactant organosilicon compound of a tantalum or this sol liquid, and the thermosetting resin and ionizing-radiation hardening mold resin that can form a rebound ace court layer gel film. As for the above-mentioned titanium or the metallic-oxide sol of a tantalum, it is desirable to form by hydrolyzing those metal alkoxides. The metal alkoxide used here is a metal alkoxide expressed with $R_mTi(OR')_n$ (R expresses the alkyl group of carbon numbers 1-10, R' expresses the alkyl group of carbon numbers 1-10, and $m+n$ is the integer of 4), or $R_mTa(OR')_n$ (R expresses the alkyl group of carbon numbers 1-10, R' expresses the alkyl group of carbon numbers 1-10, and $m+n$ is the integer of 5). Furthermore, specifically, titanium tetra-ethoxide, titanium tetra--i-propoxide, titanium tetra--n-propoxide, titanium tetra--n-butoxide, titanium tetra--sec-butoxide, titanium tetra--tert-butoxide, tantalum PENTA ethoxide, tantalum PENTA-i-propoxide, tantalum PENTA-n-propoxide, tantalum PENTA-n-butoxide, tantalum PENTA-sec-butoxide, tantalum PENTA-tert-butoxide, etc. are mentioned.

[0010] Hydrolysis of the above-mentioned metal alkoxide is performed by dissolving the above-mentioned metal alkoxide into a suitable solvent. As a solvent to be used, aromatic hydrocarbon, such as alcohol, such as a methyl ethyl ketone, isopropyl alcohol, a methanol, ethanol, methyl isobutyl ketone, ethyl acetate, and butyl acetate, a ketone, ester, halogenated hydrocarbon, toluene, and a xylene, or such mixture are mentioned, for example. By the metallic-oxide conversion produced noting that this alkoxide hydrolyzes and condenses 100% in the above-mentioned solvent, 0.1% of the weight or more, the above-mentioned alkoxide is dissolved so that it may become 0.1 - 10% of the weight preferably. If the property of a request of the functional film formed as the concentration of a metallic-oxide sol is less than 0.1 % of the weight cannot fully demonstrate but, on the other hand, exceeds 10 % of the weight, formation of transparence homogeneous membrane will become difficult. Moreover, in proportion to gel concentration, the

refractive index of the gel film obtained can be adjusted by changing metallic-oxide gel concentration to above-mentioned within the limits. Moreover, in this invention, if it is less than the above solid content, it is also possible to use together the organic substance and an inorganic substance binder.

[0011] The water more than a complement is added to the above-mentioned alkoxide solution at hydrolysis, it is 22-28 degrees C in temperature preferably, and 15-35 degrees C of churning are performed preferably for 1 to 5 hours for 0.5 to 10 hours. In this hydrolysis, it can be desirable to use a catalyst, acids, such as a hydrochloric acid, a nitric acid, a sulfuric acid, a formic acid, and an acetic acid, can be desirable as these catalysts, about 0.1-20.0 Ns of these acids can be preferably added as an about [0.5-7.0N] water solution, and the moisture in this water solution can be made into the moisture for hydrolysis. By changing the concentration of a catalyst in the above-mentioned range on the occasion of hydrolysis, the refractive index of the gel film obtained can be adjusted in proportion to the concentration of a catalyst. Like the above, the metallic-oxide sol which were obtained by carrying out is a transparent and colorless liquid, and is a stable solution whose pot life is about one month, and to the base material, its wettability is good and excellent in spreading fitness.

[0012] Furthermore, when the refractive index of the gel film obtained eventually needs to be adjusted, for example, in order to lower a refractive index, a fluorine system organosilicon compound, an organosilicon compound, a boron system organic compound, etc. can be added. Specifically A tetra-ethoxy silane, a tetramethoxy silane, tetra-propoxysilane, Tetra-butoxysilane, alkyltrialkoxysilane, the COL coat 40 (COL coat company make), Organosilicon compounds, such as MS51 (Mitsubishi Chemical make) and the Snow tex (product made from the Nissan chemistry), Who Ron FC-110,220,250 (product made from the Toagosei chemistry), SEKURARU coat A-402B (Central Glass make), Boron system compounds, such as fluorine compounds, such as heptadecafluorodecyl trimethoxysilane, tridecafluoro octyl trimethoxysilane, and trifluoropropyl trimetoxysilane, triethyl borate, trimethyl borate, boric acid TORIPUROPIRU, and tributyl borate, are mentioned. These additives may be added at the time of preparation of a sol, and may be added after formation of a sol.

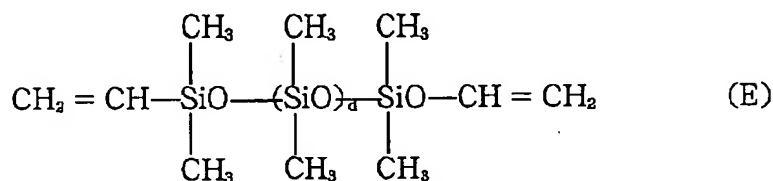
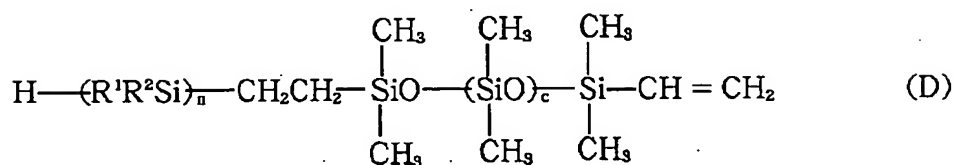
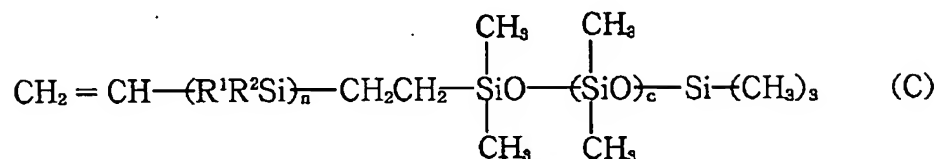
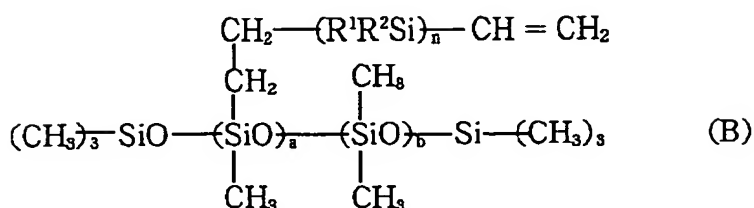
[0013] In order to gather a refractive index, a refractive index will become high, if it is necessary to change the amount or solid content concentration of the concentration of the catalyst to add, and water and each of these factors are raised. By using these additives, the time of hydrolysis of a metal alkoxide or the refractive index of the gel film with which it reacts with the hydroxyl group of gel after that, and a still more uniform and transparent sol solution is obtained and formed can be changed in a certain amount of range.

[0014] In this invention, a reactant organosilicon compound or its partial hydrolysate is added to the above-mentioned titanium or the oxide sol of a tantalum. As this reactant organosilicon compound, for example A tetramethoxy silane, A tetra-ethoxy silane, tetra--iso-propoxysilane, tetra--n-propoxysilane, Tetra--n-butoxysilane, tetra--sec-butoxysilane, tetra--tert-butoxysilane, A tetra-pentaethoxy silane, tetra-PENTA-iso-propoxysilane, Tetra-PENTA-n-propoxysilane, tetra-PENTA-n-butoxysilane, Tetra-PENTA-sec-butoxysilane, tetra-PENTA-tert-butoxysilane, Methyl trimetoxysilane, methyl triethoxysilane, a methyl tripropoxy silane, MECHIRUTORI butoxysilane, dimethyldimethoxysilane, dimethyl diethoxysilane, A dimethylethoxy silane, dimethyl methoxysilane, dimethyl propoxysilane, dimethyl butoxysilane, methyl dimethoxysilane, methyl diethoxysilane, hexyl trimethoxysilane, etc. are mentioned.

[0015] Furthermore, a with a molecular weight of 5000 or less which has two or more radicals which carry out reaction bridge formation by heat or ionizing radiation, for example, a polymerization nature double bond radical, as a desirable reactant organosilicon compound organosilicon compound is mentioned as a desirable ingredient. The vinyl organic-functions polysilane to which such a reactant organosilicon compound made piece end vinyl functionality polysilane, both-ends vinyl functionality polysilane, a piece end vinyl organic-functions polysiloxane, both-ends vinyl organic-functions polysiloxanes, or these compounds react, or a vinyl organic-functions polysiloxane is mentioned. It will be as follows if a concrete compound is illustrated.

[0016]





(a~dは1~分子量が5000以下になる値である)

[0017] If others carry out a compound, acryloxy (meta) silane compounds, such as 3-(meta)acryloxypropyltrimethoxysilane and 3-(meta)acryloxypropylmethyldimethoxysilane, are mentioned. As for the reactant organosilicon compound like ****, it is desirable to use it at a rate of the about 0.1 to 50 weight section per oxide sol (solid content) 100 weight section of said titanium or a tantalum.

[0018] The approach of forming a high refractive-index layer using the titanium containing the above-mentioned reactant organosilicon compound, or the oxide sol solution of a tantalum Whether activity energy-line exposure processing of the spreading object is carried out after that by applying this titanium or the oxide sol solution of a tantalum using the applying method to the front face of said transparence base material film or by heat-treating Being able to form titanium or the oxide sol film of a tantalum, the adhesion with the low refractive-index layer formed on it improves remarkably. As the method of application to the transparence base material film of said titanium or the oxide sol solution of a tantalum, a spin coat method, a dip method, a spray method, the roll coater method, the meniscus coating-machine method, a flexographic printing method, screen printing, the bead coating-machine method, etc. are mentioned.

[0019] Heat treatment of a spreading layer performed after spreading of the above-mentioned sol solution is performed at the temperature below the heat deflection temperature of said transparence base material film. For example, when a transparence base material film is a polyethylene terephthalate film (PET), heat treatment can be performed at the temperature of about 80-150 degrees C for about 1 minute - 1 hour, and titanium or the oxide gel film of a tantalum can be formed. What is necessary is just to determine such heat treatment conditions according to the class of transparence base material film to be used, since it changes with the classes and thickness of a transparence base material film to be used.

[0020] As an activity energy line used for hardening after spreading of a sol solution, an electron ray or ultraviolet rays

is mentioned and especially an electron ray is desirable. For example, in electron ray hardening, the electron ray which is emitted from various electron ray acceleration machines, such as a cock loft WARUTON mold, a BANDE graph mold, a resonance transformation mold, an insulating core transformer mold, a linear model, the Dynamitron mold, and a RF mold, and which has the energy of 100-300KeV preferably is used 50 to 1,000 KeV, and when it is ultraviolet curing, the ultraviolet rays emitted from the light source of an ultrahigh pressure mercury lamp, a high-pressure mercury-vapor lamp, a low pressure mercury lamp a carbon arc, a xenon arc, a metal halide lamp, etc. be used. As the total dose of an activity energy line, when an activity energy line is an electron ray, 0.5 or more Mrads are the range of 0.5 - 50Mrad preferably.

[0021] Permuting air with oxygen, as for electron beam irradiation, it is desirable to carry out in sufficient oxygen ambient atmosphere, by carrying out in an oxygen ambient atmosphere, generation of titanium or the oxide sol of a tantalum, and a polymerization and condensation are promoted, and it can form the gel layer of homogeneity and high quality more. Permuting air with oxygen, as for the above-mentioned heat treatment, it is desirable to carry out in sufficient oxygen ambient atmosphere, by carrying out in an oxygen ambient atmosphere, generation of titanium or the oxide gel of a tantalum, and a polymerization and condensation are promoted, and it can form the gel layer of homogeneity and high quality more.

[0022] Moreover, since said transparence base material film consists of thermoplastics, as for thermosetting resin and ionizing-radiation hardening mold resin which can use together with the above-mentioned sol liquid, and can form a high refractive-index rebound ace court layer, it is desirable to use the ionizing-radiation hardening mold resin which does not need an elevated temperature at the time of hardening. In addition, JIS "has hard nature" in this description What shows the degree of hardness more than H by the pencil hardness test shown by K5400 is said. [a "rebound ace court layer" or] Moreover, in this invention, the height of the relative refractive index of the layers which adjoin mutually is called a "high refractive index" and "low refractive index."

[0023] As suitable ionizing-radiation hardenability resin to form a rebound ace court layer what has the functional group of an acrylate system preferably -- for example, -- comparatively -- the polyester resin of low molecular weight -- Polyether resin, acrylic resin, an epoxy resin, urethane resin, an alkyd resin, Spiro acetal resin, a polybutadiene resin, poly thiol polyene resin, The oligomer or the prepolymers of a multifunctional compound, such as polyhydric alcohol, [, such as acrylate (meta)] As a reactant diluent, and ethyl (meta) acrylate, ethylhexyl (meta) acrylate, Monofunctional monomers, such as styrene, methyl styrene, and N-vinyl pyrrolidone, In a list polyfunctional monomer, for example, TORIMECHI roll pro pantry (meta) acrylate, Hexandiol (meta) acrylate, tripropylene GURIKORUJI (meta) acrylate, Diethylene GURIKORUJI (meta) acrylate, pen TAERISURITORUTORI (meta) acrylate, What contains dipentaerythritol hexa (meta) acrylate, 1, 6-hexane JIORUJI (meta) acrylate, neopentyl GURIKORUJI (meta) acrylate, etc. comparatively so much can be used.

[0024] Furthermore, in this invention, a with an aforementioned molecular weight of 5000 or less reactant organosilicon compound can be used as hardenability resin which forms a rebound ace court layer, mixing with the independent or above-mentioned hardenability resin. By using this organosilicon compound as some rebound ace court formation ingredients [at least], the adhesion of a rebound ace court layer and a low refractive-index layer can be raised remarkably. Furthermore, in order to use the above-mentioned ionizing-radiation hardening mold resin as ultraviolet curing mold resin, n butylamine, triethylamine, tri-n-butyl phosphine, etc. can be mixed and used as a photopolymerization initiator into this as acetophenones, benzophenones, MIHIRA benzoyl benzoate, alpha-AMIROKI SIMM ester, tetramethylthiuram monosulfide, thioxan tons, and a photosensitizer.

[0025] If crosslinking density becomes high too much at the time of hardening when forming a rebound ace court layer by the above ionizing-radiation hardenability resin independent, flexibility will fall and a crack etc. will become easy to go into a rebound ace court layer at the time of crookedness of the acid-resisting film obtained. In this case, it is desirable to mix the thermoplastics of non-reactivity to the constituent for rebound ace court stratification in an amount until it occupies about 50 % of the weight in the whole constituent. If there are too many additions of non-reactivity resin, rebound ace court nature may become imperfection.

[0026] As non-reactivity resin, thermoplastics is mainly used. When the mixture of polyester acrylate and polyurethane acrylate is especially used for ionizing-radiation hardening mold resin, polymethacrylic acid methyl acrylate or polymethacrylic acid butyl acrylate can keep the degree of hardness of a paint film high to the thermoplastics to be used. And in this case, since the refractive index with main ionizing-radiation hardening mold resin is near, the transparency of a paint film is not spoiled, but in transparency especially a low Hayes value, high permeability, and the

point of compatibility, it is advantageous.

[0027] Although the refractive index of the rebound ace court layer which consists of the above component is usually 1.49 to about 1.51, in order to raise the refractive index of a rebound ace court layer further, the ultrafine particle of the metal metallurgy group oxide of a high refractive index can be added in the resin constituent for rebound ace court stratification. The refractive indexes with a desirable rebound ace court layer are 1.50-2.10. Since the acid-resisting effectiveness is determined by the refractive index and thickness of the optical buffer film, it can heighten the acid-resisting effectiveness by adjusting both parameters. As an ingredient which has said high refractive index, impalpable powder, such as ZnO (refractive index 1.90), TiO₂ (refractive indexes 2.3-2.7), CeO₂ (refractive index 1.95), Sb₂O₅ (refractive index 1.71), SnO₂ and ITO (refractive index 1.95), Y₂O₃ (refractive index 1.87), La₂O₃ (refractive index 1.95), and ZrO₂ (refractive index 2.05), aluminum₂O₃ (refractive index 1.63), is mentioned, for example.

[0028] Moreover, in order to raise the refractive index of a rebound ace court layer further, the resin which contained the molecule and atom of a high refractive-index component in the resin constituent for rebound ace court stratification may be used. As the molecule and atom of the component which raises said refractive index, an aromatic series ring, halogen atoms other than F, the atom of S, N, and P, etc. are mentioned. After make a suitable solvent dissolve or distribute the above component, considering as coating liquid, applying this coating liquid to said base material film directly, making it harden it, or applying to a mold releasing film and stiffening it, said transparence base material film can be made to be able to imprint the rebound ace court layer which consists of the above component using suitable adhesives, and it can also be formed. The thickness of a rebound ace court layer usually has desirable about about 3-10 micrometers.

[0029] The approach of hardening by the exposure of the hardening approach of usual ionizing-radiation hardening mold resin, i.e., an electron ray, and ultraviolet rays can be used for hardening of the above-mentioned rebound ace court layer. For example, in electron ray hardening, the electron ray which is emitted from various electron ray accelerators, such as a cock loft WARUTON mold, a BANDE graph mold, a resonance transformation mold, an insulating core transformer mold, a linear model, the Dynamitron mold, and a RF mold, and which has the energy of 100-300KeV preferably is used 50 to 1000 KeV, and when it is ultraviolet curing, the ultraviolet rays emitted from beams of light, such as an ultrahigh pressure mercury lamp, a high-pressure mercury-vapor lamp, a low pressure mercury lamp, a carbon arc, a xenon arc, and a metal halide lamp, can be used

[0030] Next, the acid-resisting film of this invention is obtained by forming a low refractive-index layer in the field of the above-mentioned high refractive-index rebound ace court layer. As a low refractive-index layer, the thin film of MgF₂ of about 0.08-0.2 micrometers of thickness, or SiO₂ grade Vacuum evaporatio and sputtering, Although the approach of forming the with a refractive index of 1.44 or less SiO₂ gel film from the approach of forming by gaseous-phase methods, such as a plasma-CVD method, or the sol liquid containing SiO₂ sol obtained by hydrolyzing the aforementioned organosilicon compound in a liquid medium etc. is mentioned By forming a refractive-index layer into the above-mentioned SiO₂ sol especially from the sol liquid which added the with an aforementioned molecular weight of 5000 or less reactant organosilicon compound, the adhesion of the low refractive-index layer and rebound ace court layer which are formed improves further.

[0031] The ingredient which is the example which carried out functional separation at the rebound ace court layer 5 and the high refractive-index layer 6, and uses the rebound ace court layer which has a high refractive index in the example which shows the example shown in drawing 2 to drawing 1 in this example, the approach of forming each class, etc. are the same as that of the case of the example shown in drawing 1. As compared with the acid-resisting film shown in drawing 1, the acid-resisting film shown in drawing 2 has the low minimum reflection factor in a light field, and its acid-resisting effectiveness is large.

[0032] The example shown in drawing 3 forms the detailed irregularity configuration 7 in the front face of an acid-resisting film, and gives anti-dazzle property to an acid-resisting film. Although which a well-known approach may be used for formation of a detailed irregularity configuration, conventionally for example, as a desirable approach When forming a high refractive-index rebound ace court layer with a replica method, the mat film which has a detailed irregularity configuration is used for a front face as a base material film of imprint material. The coating liquid for high refractive-index rebound ace court layers is applied and stiffened on this film, after that, said transparence base material film plane is made to imprint this rebound ace court layer through adhesives etc. if needed, and the approach of giving a detailed irregularity configuration to the front face of a high refractive-index rebound ace court layer is mentioned. Make said base material film plane apply and dry the coating liquid for high refractive-index rebound ace court layers,

the mat film like the above is made to stick to the field of the resin layer by pressure in the condition as the other approaches, a resin layer is stiffened in the condition, subsequently a mat film is exfoliated, and the method of making the front face of a high refractive-index rebound ace court layer imprint the detailed irregularity configuration of a mat film is mentioned. Anyway, since the low refractive-index layer formed in the front face of the high refractive-index layer which has such a detailed irregularity configuration is a thin film, in the front face of a low refractive-index layer, the above-mentioned detailed irregularity configuration 7 appears.

[0033] The acid-resisting film of this invention can prepare further the layer for giving the various functions other than each class explained above. For example, in order to prepare an adhesives layer and a primer layer in order to raise the adhesion of a transparence base material film and a high refractive-index rebound ace court layer (high refractive-index layer), and to raise the hard engine performance, a rebound ace court layer can be made into two or more layers. As for the refractive index of the layer of others which are prepared in the medium of a transparence base material film and a rebound ace court layer as mentioned above, it is desirable to consider as the middle value of the refractive index of a transparence base material film and the refractive index of a rebound ace court layer.

[0034] As mentioned above, on a transparence base material film, the formation approach of a layer besides the above may apply desired coating liquid directly or indirectly, and may form it. moreover, in forming a rebound ace court layer by imprint on a transparence base material film The coating liquid which forms other layers is applied and formed on the rebound ace court layer beforehand formed on the mold releasing film. Then, other layers may be imprinted on a transparence base material film by ****ing the spreading side of a mold releasing film inside, laminating a transparence base material film and a mold releasing film, and subsequently exfoliating a mold releasing film. Moreover, the binder may be applied to the underside of the acid-resisting film of this invention, and this acid-resisting film can be stuck and used for the object which should be carried out acid resisting, for example, a polarizing element, on it.

[0035] The acid-resisting film of this invention which are obtained by carrying out is [the front face of the polarizing plate used for various displays, such as a word processor, a computer, television, and a plasma display panel, and a liquid crystal display, the sunglass lens which consists of transparent plastics, and whenever] useful like the above to acid resisting of front faces, such as windowpanes, such as covering of optical lenses, such as a with glass lens and a finder lens for cameras, and various instruments, an automobile, and an electric car.

[0036]

[Example] Next, an example and the example of a comparison are given and this invention is explained still more concretely.

It agitated for 30 minutes until it dissolved tetrabutoxytitanium in the ethylcellosolve which is a solvent and solution temperature was stabilized at 25 degrees C so that solid content concentration when example 1 tetrabutoxytitanium (Ti (OR₄)) assumes ideally that it hydrolyzed and condensed in TiO₂ might become 3 % of the weight (A liquid). In A liquid, hydrolysis was performed for the hydrochloric acid with a concentration of 3 Ns which is a catalyst at molar quantity **** and 25 degrees C 2.5 times to the alkoxide radical of tetrabutoxytitanium for 3 hours, and the TiO₂ sol solution was obtained (B liquid).

[0037] In the above-mentioned B liquid, the with a molecular weight of 5000 or less vinyl group content silane (X-12-2400: a trade name, Shin-Etsu Chemical make) was added at a rate of 5 weight sections to the B liquid 100 weight section (3% of solid content), and the coating liquid for high refractive-index layers (refractive index 1.9) was obtained. a PET film (T-60: -- a trade name and diamond foil incorporated company make --) with a smooth front face 50-micrometer top in thickness -- ionizing-radiation hardening mold FOSUFAZEN denaturation acrylate (PPZ-N-2000: -- a trade name --) Coating of the liquefied resin constituent which blended the product made from the Osaka Kyoeisha chemistry and the above-mentioned coating liquid for high refractive-index layers with the weight ratio 2:1 is carried out with a gravure reverse coat so that it may become 7 micrometer/dry. After carrying out 5Mrad exposure of the electron ray with the acceleration voltage of 175kV, heat-treat for 1 hour, it was made to harden at 120 degrees C, and the high refractive-index rebound ace court layer (refractive index 1.75) was formed.

[0038] On this high refractive-index rebound ace court layer, coating of the 2 liquid hardening mold adhesives (LX660 (base resin), KW75 (curing agent): a trade name, Dainippon Ink & Chemicals make) was carried out with the gravure reverse coat, and the adhesives layer was formed. Subsequently, after laminating a PET film (A-4300 : 100 micrometers in a trade name, the Toyobo make, thickness) through this adhesives layer and aging for four days at 40 degrees C, the above-mentioned PET film (T-60) was exfoliated, and the high refractive-index rebound ace court layer

was made to imprint on a PET film (A-4300).

[0039] On the high refractive-index rebound ace court layer on the obtained PET film (A-4300), SiOx was further vapor-deposited by the plasma-CVD method, the SiOx layer (refractive index 1.46) of 100nm of thickness was formed, and the acid-resisting film of this invention was obtained. 94.0%, the Hayes value 0.5 and the minimum reflection factor in the wavelength field of a visible ray are 0.4, and the total light transmission of the obtained acid-resisting film excelled [reflection factor] in acid resistibility. Moreover, the surface pencil degree of hardness is 3H, and was excellent also in the rebound ace court engine performance. Furthermore, the adhesion of a high refractive-index rebound ace court layer and a low refractive-index layer is 100/100 in a squares tape friction test, and was excellent also in adhesion.

[0040] It agitated for 30 minutes until it dissolved MTEOS in the methyl ethyl ketone which is a solvent and solution temperature was stabilized at 25 degrees C so that solid content concentration when example 2 methyl triethoxysilane (MTEOS) assumes ideally that it hydrolyzed and condensed in SiO₂ or MeSiO_{1.5} might become 3 % of the weight. Into this solution, hydrolysis was performed for the hydrochloric acid with a concentration of 0.005 Ns which is a catalyst at the alkoxide radical of MTEOS, equimolecular amount ****, and 25 degrees C for 3 hours. What mixed sodium acetate and an acetic acid as a curing agent further was added to this solution, it agitated at 25 degrees C for 1 hour, and the SiO₂ sol solution was obtained. In the above-mentioned solution, the with a molecular weight of 5000 or less vinyl group content silane (X-12-2400: trade name) was added at a rate of 10 weight sections to the SiO₂ sol liquid 100 weight section (3% of solid content), and the coating liquid for low refractive-index layers was obtained.

[0041] the mat PET film (lumiler E-06 and :trade name --) with which detailed irregularity is formed in the front face Coating of the liquefied resin constituent which blended ionizing-radiation hardening mold FOSUFAZEN denaturation acrylate (PPZ-N -2000: trade name), an example 1, and the same coating liquid for high refractive-index layers on 50 micrometers in the Toray Industries make and thickness at the weight ratio 2:1 is carried out with a gravure reverse coat so that it may become 7 micrometer/dry. After carrying out 5Mrad exposure of the electron ray with the acceleration voltage of 175kV, heat-treat for 1 hour, it was made to harden at 120 degrees C, and the high refractive-index anti-dazzle property rebound ace court layer was formed (refractive index 1.65). Subsequently, on this high refractive-index rebound ace court layer, coating of the 2 liquid hardening mold adhesives (LX660 (base resin), KW75 (curing agent): trade name) was carried out with the gravure reverse coat, and the adhesives layer was formed. Subsequently, after laminating a PET film (A-4300) through this adhesives layer and aging for four days at 40 degrees C, the above-mentioned PET film (lumiler E-06) was exfoliated, and the high refractive-index anti-dazzle property rebound ace court layer was made to imprint on a PET film (A-4300). The front face of this high refractive-index anti-glare layer serves as the shape of surface type of said PET film (lumiler E-06), and the same detailed concavo-convex configuration.

[0042] On the high refractive-index rebound ace court layer on the obtained PET film (A-4300), coating of the aforementioned coating liquid for low refractive-index layers (refractive index 1.42) was carried out so that the thickness after desiccation might be set to 0.1 micrometers, heat treatment of 1 hour was performed at 120 degrees C, and the anti-dazzle property acid-resisting film of this invention was obtained. 92.5%, the total light transmission of the obtained acid-resisting film is the Hayes value 1.0, and was excellent in acid resistibility and anti-dazzle property. Moreover, the surface pencil degree of hardness is 3H, and was excellent also in the rebound ace court engine performance. Furthermore, the adhesion of a high refractive-index rebound ace court layer and a low refractive-index layer is 100/100 in a squares tape friction test, and was excellent also in adhesion.

[0043] The PET film (lumiler T-60: trade name) with a thickness of 50 micrometers was prepared as an example 3 transparence base material film. On the other hand, the coating liquid for high refractive-index layers of an example 1 (refractive index 1.9) and ionizing-radiation hardening mold resin were blended so that it might become the ionizing-radiation hardening mold resin 1 weight section to the titanic-acid ghost sol 15 weight section in this coating liquid. Coating was carried out with the gravure reverse coat so that it might become thickness 0.1 micrometer/dry after drying this liquefied resin constituent on said PET film, heat treatment was performed at 120 degrees C for 1 hour, and the high refractive-index layer (refractive index 1.9) was formed. On this high refractive-index layer, coating of the rebound ace court resin (PPZ-N -2000: trade name) was carried out with the gravure reverse coat so that it might become 7 micrometer/dry, 5Mrad exposure of the electron ray was carried out with the acceleration voltage of 175kV, the paint film was stiffened, and the rebound ace court layer was formed (refractive index 1.55).

[0044] On this high refractive-index rebound ace court layer, coating of the 2 liquid mold-curing mold adhesives (LX660 (base resin), KW75 (curing agent): trade name) was carried out with the gravure reverse coat, and the

adhesives layer was formed. Subsequently, after laminating a PET film (A-4300: trade name) through this adhesives layer and aging for four days at 40 degrees C, the above-mentioned PET film (T-60) was exfoliated, and the high refractive-index anti-dazzle property rebound ace court layer was made to imprint on a PET film (A-4300).

[0045] On the high refractive-index rebound ace court layer on the obtained PET film (A-4300), SiO_x was further vapor-deposited by the plasma-CVD method, the SiO_x layer (refractive index 1.46) of 0.1 micrometers of thickness was formed, and the acid-resisting film of this invention was obtained. 94.0%, the total light transmission of the obtained acid-resisting film is the Hayes value 0.5, and was excellent in acid resistibility and anti-dazzle property. Moreover, the surface pencil degree of hardness is 3H, and was excellent also in the rebound ace court engine performance. Furthermore, the adhesion of a high refractive-index rebound ace court layer and a low refractive-index layer is 100/100 in a squares tape friction test, and was excellent also in adhesion.

[0046] In the example 4 aforementioned example 3, on the rebound ace court layer (refractive index 1.9) of the high refractive index currently formed on the PET film (A-4300), coating of the coating liquid for low refractive-index layers of said example 2 (refractive index 1.42) was carried out to the business from which the thickness after desiccation is set to 0.1 micrometers, heat treatment of 1 hour was performed at 120 degrees C, and the acid-resisting film of this invention was obtained. The total light transmission of the obtained acid-resisting film was 94.5% and the Hayes value 0.4, and the minimum reflection factor in the wavelength field of a visible ray is 0.2, and was excellent in acid resistibility. Moreover, the surface pencil degree of hardness is 3H, and was excellent also in the rebound ace court engine performance. Furthermore, the adhesion of a high refractive-index rebound ace court layer and a low refractive-index layer is 100/100 in a squares tape friction test, and was excellent also in adhesion.

[0047] On the PET film (A-4300: trade name) with example 5 smooth front face, coating of the liquefied resin constituent which blended said coating liquid for high refractive-index layers and rebound ace court resin (PPZ-N - 2000: trade name) with the weight ratio 2:1 was carried out with the gravure reverse coat so that it might become 7 micrometer/dry, and desiccation clearance of the solvent was carried out. Subsequently, the mat PET film (lumiler E-06 : 50 micrometers in a trade name, thickness) with which detailed irregularity is formed in the front face through this high refractive-index rebound ace court layer was laminated, 5Mrad exposure of the electron ray was carried out with the acceleration voltage of 175kV, the paint film was stiffened, the above-mentioned PET film (lumiler E-06) was exfoliated, and the high refractive-index anti-dazzle property rebound ace court layer was formed. The front face of the high refractive-index anti-dazzle property rebound ace court layer on this PET film (A-4300) serves as the shape of surface type of said PET film (lumiler E-06), and the same detailed concavo-convex configuration.

[0048] On the high refractive-index rebound ace court layer on the obtained PET film (A-4300), SiO_x was further vapor-deposited by the plasma-CVD method, the SiO_x layer (refractive index 1.46) of 0.1 micrometers of thickness was formed, and the acid-resisting film of this invention was obtained. 92.5%, the total light transmission of the obtained acid-resisting film is the Hayes value 1.0, and was excellent in acid resistibility and anti-dazzle property. Moreover, the surface pencil degree of hardness is 3H, and was excellent also in the rebound ace court engine performance. Furthermore, the adhesion of a high refractive-index rebound ace court layer and a low refractive-index layer is 100/100 in a squares tape friction test, and was excellent also in adhesion.

[0049] Coating was carried out so that the thickness after drying the low refractive-index layer coating liquid (refractive index 1.42) of said example 2 on the anti-dazzle property high refractive-index rebound ace court layer of the example 6 aforementioned example 4 might be set to 0.1 micrometers, heat treatment of 1 hour was performed at 120 degrees C, and the acid-resisting film of this invention was obtained. 93.5%, the total light transmission of the obtained acid-resisting film is the Hayes value 1.0, and was excellent in acid resistibility and anti-dazzle property. Moreover, the surface pencil degree of hardness is 3H, and was excellent also in the rebound ace court engine performance. Furthermore, the adhesion of a high refractive-index rebound ace court layer and a low refractive-index layer is 100/100 in a squares tape friction test, and was excellent also in adhesion.

[0050] It agitated for 30 minutes until it dissolved in the isopropyl alcohol (IPA) which is a solvent and solution temperature was stabilized at 25 degrees C so that solid content concentration when an example 7 pentaethoxy tantalum (PEOT) assumes ideally that it hydrolyzed and condensed in Ta 2O₅ might become 3 % of the weight. In the above-mentioned solution, the one mol pair of the water which dissolved the hydrochloric acid with a concentration of 1 N which is a catalyst of PEOT(s) was carried out, in addition, hydrolysis was performed at the room temperature for 3 hours, and 5 mols of sol liquid were obtained. The with a molecular weight of 5000 or less vinyl group content silane (X-12-2400: trade name) was added at a rate of 5 weight sections to the sol liquid-solid form part 100 weight section in

this sol liquid, and the coating liquid for high refractive-index layers (refractive index 1.9) was obtained in it. The acid-resisting film of this invention was obtained like the example 1 except having used this coating liquid for high refractive-index layers. This acid-resisting film had the adhesion of the total light transmission, the Hayes value, the acid resistibility, the surface pencil degree of hardness and high refractive-index rebound ace court layer, and low refractive-index layer which were excellent like the case of an example 1.

[0051] In the acid-resisting film of the example of comparison 1 aforementioned example 1, the acid-resisting film was produced like the example 1 except having formed the refractive-index layer using the TiO₂ sol solution which does not add a reactant silicon organic compound (X-12-2400). The total light transmission of this acid-resisting film was 94.5% and the Hayes value 0.5, and although acid resistibility was the same as that of said example 1, the adhesion of a high refractive-index rebound ace court layer and a low refractive-index layer was 80/100 in the squares tape friction test.

[0052] In the acid-resisting film of the example of comparison 2 aforementioned example 1, the acid-resisting film was produced like the example 1 except having used as the high refractive-index layer the TiO_x film formed with the vacuum deposition method. The total light transmission of this acid-resisting film is 93.7% and the Hayes value 0.7, and acid resistibility is falling as compared with said each example. Moreover, the surface pencil degree of hardness was 2H, and the adhesion of a high refractive-index rebound ace court layer and a low refractive-index layer was 60/100 in the squares tape friction test.

[0053]

[Effect of the Invention] According to this invention, like the above, it can provide economically by forming the high refractive-index rebound ace court layer of an acid-resisting film as with a refractive indexes of 1.65 or more gel film into a liquid medium from the sol liquid containing titanium, or the oxide sol and the reactant organosilicon compound of a tantalum, without using the acid-resisting film which has the adhesion in which the low refractive-index layer was excellent to the high refraction rebound ace court layer for an expensive and complicated facility etc.

[Translation done.]